

(19) Japan Patent
Office (JP)

(12) Japanese
Patent Laid-Open
Application
Publication (A)

(11) Patent Laid-Open
Application
No. 5-43402

(43) Date of publication
of application:
February 23, 1993

(51) Int.Cl. ⁵	Identification Symbol	Internal File No.	FI	Technical Field
A 01 N	25/14	6742-4H		
	25/04	6742-4H		
	25/30	6742-4H		

Number of Claims: 3

Request for Examination: Unrequested
(5 pages in all)

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(54) [Title of the Invention]

Water-dispersible granular agrochemical composition

(57) [Abstract]

[Constitution]

A water-dispersible granular agrochemical composition,
characterized in that, the composition contains one or more

agrochemical active ingredients, the following surfactant (1) and the following surfactant (2):

(1) a surfactant such as ligninsulfonic acid and a condensate of naphthalenesulfonic acid with formalin;

(2) a surfactant such as alkylnaphthalenesulfonic acid and polyoxyethylenealkyl phenyl ether sulfate.

[Effect]

The agrochemical composition of the present invention has excellent disintegrating property and dispersing property in water.

[Claims for the Patent]

[Claim 1]

A water-dispersible granular agrochemical composition, characterized in that, the composition comprises one or more of an agrochemical active ingredients, the following surfactant (1) and the following surfactant (2):

(1) one or more surfactants selected from ligninsulfonic acid, a condensate of naphthalenesulfonic acid with formalin, a copolymer of maleic acid with diisobutylene and a copolymer of maleic acid with isobutylene, and alkali metal salt, alkali earth metal salt, ammonium salt and amine salt thereof;

(2) one or more surfactants selected from alkylnaphthalenesulfonic acid, polyoxyethylenealkyl phenyl ether sulfate and polyoxyethylenestyryl phenyl ether sulfate, and alkali metal salt, alkali earth metal salt, ammonium salt and amine salt thereof.

[Claim 2]

The water-dispersible granular agrochemical composition as claimed in claim 1, wherein the composition comprises from 5 to 30% by weight of the surfactant(s) (1).

[Claim 3]

The water-dispersible granular agrochemical composition as claimed in claim 1, wherein the composition comprises from 1 to 10% by weight of the surfactant(s) (2).

[Detailed Description of the Invention]

[0001]

[Industrial Application Field]

The present invention relates to a water-dispersible granular agrochemical composition having excellent disintegrating property and dispersing property in water.

[0002]

[Conventional Art]

Examples of the preparations which are sprinkled by diluting with water among agrochemical preparations are emulsion, hydrating agent, flowable agent and the like. Since the emulsion uses an organic solvent as a carrier, there are problems of toxicity, irritation, ignition, smell and the like. On the other hand, the hydrating agent is a finely powdery preparation and, therefore, its measurement is not convenient and there is a problem of flying of the powder during the preparation of a spraying liquid. The flowable agent is a preparation where a preparation in a suspended state is produced whereby the inconvenience in measurement and the problem of flying of powder are solved but, since it is a viscous liquid preparation, it is hardly taken out from a container and, since a small amount of the preparation remains in the container, discarding of the container is sometimes difficult.

[0003]

In view of the above, attempts for making the hydrating agent into granules have been conducted in recent years. As a result of making the hydrating agent granules, the flying of powder and the difficulty in measurement are improved. Further, there is also no problem in handling caused by the high viscosity of a flowable agent. Various proposals have been done concerning the technique for the manufacture of granulated

hydrating agent as above (the above water-dispersible granular agrochemical composition) already. They are, for example, a method where starch and a water-soluble inorganic salt are compounded (Japanese Patent Publication No. 53-12577), a method where saccharide, a surfactant of a naphthalenesulfonic acid type and alkali metal phosphate are compounded (Japanese Patent Laid-Open No. 57-163303) and a method where anionic surfactant of a sulfate type and nonionic surfactant are combined and compounded (Japanese Patent Laid-Open No. 59-193803).

[0004]

[Problems to be Solved by the Invention]

However, the granulated hydrating agent as such does not give good disintegrating property and dispersing property in water when an active ingredient as the agrochemical is contained in high concentrations and, further, its disintegrating property and dispersing property in water are deteriorated with age during storage whereby the agent is not always practical.

[0005]

[Means for Solving the Problems]

The present inventor has carried out intensive studies for solving the above-mentioned problems and, as a result, has found a method where, even when the active component as agrochemical is contained in high concentrations, good disintegrating property and dispersing property in water are achieved and, during storage, the disintegrating and dispersing properties in water are not deteriorated with age, whereby the present invention has been accomplished.

[0006]

Thus, the present invention relates to a water-dispersible granular agrochemical composition, characterized in that, the composition contains one or more of agrochemical active ingredients, the following surfactant (1) and the following surfactant (2). With regard to the surfactant (1), there may be used one or more surfactants selected from ligninsulfonic acid, a condensate of naphthalenesulfonic acid with formalin, a copolymer of maleic acid with diisobutylene and a copolymer of maleic acid with isobutylene, and alkali metal salt, alkali earth metal salt, ammonium salt and amine salt thereof. With regard to the surfactant (2), there may be used one or more surfactants selected from alkylnaphthalenesulfonic acid, polyoxyethylenealkyl phenyl ether sulfate and polyoxyethylenestyryl phenyl ether sulfate, and alkali metal salt, alkali earth metal salt, ammonium salt and amine salt thereof.

[0007]

It is preferred that, in the agrochemical composition, the content of the surfactant (1) is preferably from 5 to 30% by weight and, more preferably, from 10 to 20% by weight. The content of the surfactant (2) is preferably from 1 to 10% by weight and, more preferably, from 2 to 5% by weight. There is no particular limitation for the active component as agrochemical used in the present invention and such an agent which has been used for a hydrating agent may be applied. Representative examples thereof will be listed below.

[0008]

Thus, examples of an insecticide include 2-tert-butyl-5-(4-tert-butylbenzylthio)-4-chloropyridazin-3(2H)-one (generic name: pyridaben), 1-naphthyl-N-methyl carbamate (generic name: NAC), 3,7,9,13-tetramethyl-5,11-dioxo-2,8,14-trithia-4,7,9,12-tetraazapentadeca-3,12-dien-6,10-dione (generic name: thiodicarb), 3-methyl-1,5-bis(2,4-xylyl)-1,3,5-triazapenta-1,4-diene (generic name: amitraz), 3,6-bis(2-chlorophenyl)-1,2,4,5-tetrazine (generic name: clofentezine), hexakis(β,β -dimethylphenethyl)-distannoxane (generic name: fenbutatin oxide) and isopropyl 4,4'-dibromobenzylate (generic name: phenisobromolate). Examples of a bactericide include 2,4'-dichloro- α -(pyrimidin-5-yl)benzhydryl alcohol (generic name: Fenarimol), 8-hydroxyquinoline copper (generic name: oxine copper), 5-methyl-1,2,4-triazolo[3,4-b]benthiazole (generic name: tricyclazole), 3-(3,5-dichlorophenyl)-N-isopropyl-2,4-dioxoimidazolidine-1-carboxamide (generic name: iprodione), N-trichloromethylthiotetrahydrophthalimide (generic name: captan) and 2,6-dichloro-4-nitroaniline (generic name: CNA). Examples of a herbicide include ethyl 5-(4,6-dimethoxypyrimidin-2-ylcarbamoylsulfamoyl)-1-methylpyrazole-4-carboxylate (generic name: pyrazosulfuron ethyl), methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate, ethyl (RS)-2-[4-(6-chloroquinoxalin-2-yloxy)phenoxy]propionate (generic name: quizalofop-ethyl), 2-chloro-2',6'-diethyl-N-(2-propoxyethyl)acetanilide (generic name: pretilachlor), ammonium DL-homoalanin-4-yl (methyl)phosphinate (generic name: glufosinate), 5-tert-butyl-3-(2,4-dichloro-5-isopropoxyphenyl)-1,3,4-oxadiazolin-2-one

(generic name: oxadiazon), methyl N-(3,4-dichlorophenyl)carbamate (generic name: MCC), 3-isopropyl-2,1,3-benzo-thiadiazinone-(4)-2,2-dioxide (generic name: bentazon), 2,4-dichlorophenoxyacetic acid (generic name: 2,4-D), 2-methyl-4-chlorophenoxyacetic acid (generic name: MCP), 2-methylthio-4,6-bis(isopropylamino)-s-triazine (generic name: prometryn), 2-methylthio-4-ethylamino-6-isopropylamino-s-triazine (generic name: ametryn), 2-chloro-4,6-bis(ethylamino)-s-triazine (generic name: simazine), 2-chloro-4-ethylamino-6-isopropylamino-s-triazine (generic name: atrazine) and 2-methylthio-4,6-bis(isopropylamino)-s-triazine (generic name: prometryn). Depending upon the purpose, the agrochemical active ingredient may be used either solely or jointly by combining two or more thereof.

[0009]

In the agrochemical composition of the present invention, the amount of the agrochemical active ingredient is preferably from 1 to 90% by weight and, more preferably, from 10 to 80% by weight. If necessary, the granulated hydrating agent of the present invention (the above-mentioned water-dispersible granular agrochemical composition) may further contain mineral powder or water-soluble powder as an extender. With regard to the mineral powder, there may be used diatomaceous earthy, talc, clay, bentonite, calcium carbonate and the like. With regard to the water-soluble powder, there may be used saccharide, urea, various kinds of salt and the like. Examples of the saccharide are lactose, fructose, glucose and the like, while examples of the salt are alkali metal salt, ammonium salt and the like of

sulfuric acid, phosphoric acid, hydrochloric acid, nitric acid and carbonic acid. Such an extender may be used solely or two or more thereof may be mixed.

[0010]

If necessary, binder, auxiliary disintegrating agent, absorber, degradation preventer, coloring agent, defoaming agent and the like may be also added thereto as other adjuvant. The granulated hydrating agent according to the present invention is manufactured by the following methods. The first manufacturing method is that necessary amounts of agrochemical active ingredient, surfactant (1), surfactant (2), extender and other excipients are added, homogeneously mixed and after that, finely ground. The fine grinding is able to be carried out by a dry grinder such as a shock grinder, a ball mill or a Jet-O-Mizer. After that, an appropriate amount of water is added to the resulting fine powder, mixed, kneaded, granulated using a granulating machine and dried to give a desired product. Granulation is able to be carried out using a granulating machine such as an extruding granulator, a compressing granulator, a fluidized-bed granulator, a stirring granulator and a tumbling granulator.

[0011]

The second manufacturing method is that necessary amounts of agrochemical active ingredient, surfactant (1), surfactant (2), extender and other excipients are added to an appropriate amount of water and finely ground using a wet grinder. The fine grinding is able to be carried out using a wet grinder such as a ball mill and a sand grinder. After that, appropriate amounts of

surfactant (1), surfactant (2), extender and other excipients are further added, if necessary, to the resulting aqueous suspension, mixed and spray-dried using a spray-drier to give a desired product.

[0012]

The third manufacturing method is that necessary amounts of agrochemical active ingredient, surfactant (1), surfactant (2), extender and other excipients are added to an appropriate amount of water and finely ground using a wet grinder. The fine grinding is able to be carried out using a wet grinder such as a ball mill and a sand grinder. After that, an extender is added to the resulting aqueous suspension together, if necessary, with appropriate amounts of surfactant (1), surfactant (2) and other excipients and the resulting pasty product is mixed, kneaded, granulated using a granulator and dried to give a desired product. Granulation is able to be carried out using a granulating machine such as an extruding granulator, a compressing granulator, a fluidized-bed granulator, a stirring granulator and a tumbling granulator.

[0013]

Although there is no particular limitation for the particle size of the granulated hydrated agent of the present invention, it is preferred to be of from 0.1 to 2 mm.

[0014]

[Examples]

Now the present invention will be illustrated by way of the specific Examples of the present invention as follows although

the present invention is not limited thereto. The term part(s) stands for part(s) by weight in all of the Examples.

Example 1

97 parts of pyridaben (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 70 parts of the resulting finely ground product, 10 parts of a mixture of maleic acid/diisobutylene copolymer with polyoxyethylenestyryl phenyl ether sulfate salt, 2.5 parts of ammonium polyoxyethylene alkyl phenyl ether sulfate, 5 parts of ammonium sulfate and 12.5 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Example 2

97 parts of pyrazosulfuron ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 10 parts of a mixture of maleic acid/diisobutylene copolymer with polyoxyethylenestyryl phenyl ether sulfate salt, 2.5 parts of ammonium polyoxyethylene alkyl phenyl ether sulfate, 5 parts of ammonium sulfate and 7.5 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50

meshes to give a granular hydrated agent of the present invention.

Example 3

97 parts of pyrazosulfuron ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of a condensate of sodium naphthalenesulfonate with formalin, 3 parts of sodium alkylnaphthalenesulfonate and 15 parts of clay were mixed using a mixer, further mixed with 15 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Example 4

97 parts of pyrazosulfuron ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of a condensate of sodium naphthalenesulfonate with formalin, 2 parts of sodium ligninsulfonate, 2 parts of sodium alkylnaphthalenesulfonate and 14 parts of clay were mixed using a mixer, further mixed with 15 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Example 5

97 parts of quizalofop-ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 10 parts of a mixture of maleic acid/diisobutylene copolymer with polyoxyethylenestyryl phenyl ether sulfate salt, 2.5 parts of ammonium polyoxyethylene alkyl phenyl ether sulfate, 5 parts of ammonium sulfate and 7.5 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Example 6

55 parts of pyridaben (generic name), 2 parts of a mixture of maleic acid/diisobutylene copolymer with polyoxyethylenestyryl phenyl ether sulfate salt and 43 parts of water were mixed with a homo-mixer and ground using a sand grinder (manufactured by Aimex KK) for 90 minutes to give a ground slurry. 74.9 parts of the resulting ground slurry, 13.5 parts of a mixture of maleic acid/diisobutylene copolymer with polyoxyethylenestyryl phenyl ether sulfate salt, 2.5 parts of ammonium polyoxyethylene alkyl phenyl ether sulfate, 10 parts of ammonium sulfate and 31.3 parts of clay were mixed using a mixer and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50

meshes to give a granular hydrated agent of the present invention.

Example 7

97 parts of methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]- carbonyl]amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4- carboxylate and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of a condensate of sodium naphthalenesulfonate with formalin, 2 parts of sodium ligninsulfonate, 2 parts of sodium alkyl naphthalenesulfonate and 14 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Comparative Example 1

97 parts of pyridaben (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 70 parts of the resulting finely ground product, 10 parts of a maleic acid/diisobutylene copolymer, 5 parts of ammonium sulfate and 15 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50

meshes to give a granular hydrated agent of the present invention.

Comparative Example 2

97 parts of pyrazosulfuron ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 10 parts of a maleic acid/diisobutylene copolymer, 5 parts of ammonium sulfate and 10 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Comparative Example 3

97 parts of pyrazosulfuron ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of a condensate of sodium naphthalenesulfonate with formalin and 18 parts of clay were mixed using a mixer, further mixed with 15 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Comparative Example 4

97 parts of quizalofop-ethyl (generic name) and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of ammonium polyoxyethylene alkyl phenyl ether sulfate, 5 parts of ammonium sulfate and 13 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

Comparative Example 5

97 parts of methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]-carbonyl]amino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate and 3 parts of white carbon were finely ground using a Jet-O-Mizer (manufactured by Seishin Enterprise Co., Ltd.). 75 parts of the resulting finely ground product, 7 parts of sodium alkylnaphthalenesulfonate and 18 parts of clay were mixed using a mixer, further mixed with 30 parts of water and granulated using an extrusion granulator equipped with a screen of 0.5 mm diameter. The granules were dried at 50°C and subjected to a particle size selection using a sieve of 20 to 50 meshes to give a granular hydrated agent of the present invention.

[0015]

Now, a test for disintegration in water and a test for stability of suspension were conducted for the preparations manufactured in Examples and Comparative Examples.

Test Example 1

Test for disintegration in water

1.0 g of a sample was precisely weighed. The weighed sample was poured into a 100-ml graduated cylinder with ground stopper in which 100 ml of hard water (3°) of 20°C was placed. After being allowed to stand for 20 seconds, the graduated cylinder was turned upside down at the rate of one turn every two seconds and the turned numbers until the granulated hydrated agent was completely disintegrated were adopted as a degree of disintegrating property in water. The less the turned numbers, the better. The result is shown in Table 1.

Test Example 2

Test for stability of suspension

1.0 g of a sample was precisely weighed in a 100-ml beaker, 50 ml of hard water (3°) of 20°C was added and the mixture was well mixed and dispersed. It was transferred to a 250-ml cylinder with a stopper and made into 250 ml by adding of hard water (3°) of 20°C and the mixture was allowed to stand for 15 minutes, vigorously shaken for 30 times a minute and allowed to stand for 5 minutes. After that, a 25-ml hole pipette was placed in the liquid, the front end thereof was placed in the center of the liquid, 25 ml of the test liquid was taken thereinto quietly, dry weight (residue after drying) in the test liquid was weighed and a suspending rate was calculated by the following formula.

[0016]

$$\text{Suspending Rate (\%)} = [(B \times 10)/A] \times 100$$

A: weight of the firstly precisely weighed sample

B: weight of the residue after drying in the test liquid

With regard to the suspending rate, the more, the better and the result is shown in Table 1.

Table 1

	Disintegrating Property in Water (times)	Suspending Rate (%)
Example 1	5	94
Example 2	3	98
Example 3	3	97
Example 4	3	98
Example 5	3	93
Example 6	6	92
Example 7	3	98
Comparative Example 1	20 or more	25
Comparative Example 2	20 or more	31
Comparative Example 3	20 or more	43
Comparative Example 4	20 or more	58
Comparative Example 5	20 or more	62

[0017]

[Advantages of the Invention]

The composition of agrochemical according to the present invention has excellent disintegrating property and dispersing property in water.